भारतीय मानक
Indian Standard

IS 16146 : 2014
(Reaffirmed 2020)

सनसेट येलो की एल्युमिनियम लेक, एफ सी एफ, खाद्य ग्रेड — विशिष्टि

Aluminium Lake of Sunset Yellow, FCF, Food Grade — Specification

ICS 67.220.20

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भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली–110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.org.in www.standardsbis.in

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Food Additives Sectional Committee, FAD 8

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

With the increased production of processed foods, manufacturers have started adding a large number of substances generally in small quantities, to improve the appearance, flavour, texture or storage properties of the processed foods. As impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives.Aluminium Lake of Sunset Yellow FCF is used as colouring agent in orange squash, orange jelly, marzipan, Swiss roll, apricot jam, citrus marmalade, lemon curd, sweets, hot chocolate mix and packet soups, trifle mix, cereals, baked goods, snack foods, ice cream, beverages, dessert powders, confectionery, etc.

This standard has been prepared to cover the requirements for Aluminium Lake of Sunset Yellow FCF food grade. This standard covers the requirements, methods of sampling and test for food grade. Aluminium Lake of Sunset Yellow FCF is used as colouring agent. Aluminium Lake is a fine orange yellow water soluble, odourless powder. It is prepared by precipitating Sunset Yellow FCF (conforming to requirements specified) on to a substratum of Alumina. Aluminium lakes are prepared under aqueous conditions by reacting aluminium oxide with colouring matter complying with purity criteria. Undried aluminium oxide is usually freshly prepared by reacting aluminium sulphate or aluminium chloride with sodium carbonate or sodium bicarbonate or aqueous ammonia. Following lake formation, the product is filtered, washed with water and dried. Unreacted aluminium oxide may also be present in the final product.

This standard is based on joint FAO/WHO expert Committee on Food Additives (JECFA) standard. In the preparation of this standard, due consideration has been given to the *Food Safety and Standards Act* 2006 and the Rules framed thereunder. Due consideration has also been given to the *Legal Metrology (Packaged Commodities) Rules*, 2011. However, this standard is subject to restrictions imposed under these, wherever applicable.

Description

Common Name — Aluminum Lake of Sunset Yellow FCF.

Chemical Name — Sunset Yellow FCF Aluminium Lake -6, hydroxy-5 (4-sulphophenlyazo)-2 Naphthalene sulphonic acid, Aluminium Lake.

Synonym — CI Pigment Yellow, 104, FD and C Yellow No. 6, Aluminium Lake (USA), Food Yellow No. 5 Aluminium Lake (Japan).

Empirical formula — $C_{16}H_{10}N_2O_7S_2Al_2$

Molecular weight — 460

Solubility — Insoluble in most organic solvents like alcohols, acetone and also in water in *p*H 3.5 to 7.5. But outside this range Lake Substrate tends to dissolve by releasing the captive dyes.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ALUMINIUM LAKE OF SUNSET YELLOW, FCF, FOOD GRADE — SPECIFICATION

1 SCOPE

This standard prescribes the requirement and the methods of sampling and test for Aluminum Lake of Sunset Yellow FCF, food grade.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
264 : 2005	Nitric acid — Specification (*third revision*)
1070 :1992	Regent grade water (*third revision*)
1695 : 2014	Sunset yellow FCF, food grade — Specification (*third revision*)
1699 : 1995	Methods of sampling and test for food colours (*second revision*)
2491 : 2013	Code for hygienic conditions for food processing units (*third revision*)

3 REQUIREMENTS

3.1 Description of Aluminium Lake of Sunset Yellow

Aluminium lake of Sunset yellow is a fine orange yellow water soluble, odourless powder, prepared by precipitating Sunset Yellow FCF, food grade (conforming to IS 1695) on to a substratum of alumina.

3.2 Solubility

Aluminium lake of Sunset yellow is insoluble in most organic solvents like alcohols, acetone and also in water in *p*H 3.5 to 7.5. But outside this range Lake Substrate tends to dissolve by releasing the captive dyes.

3.3 Freedom from Contaminants

Precaution shall be taken to ensure that the material is free from mercury, copper and chromium in any form; aromatic amines, aromatic nitro compounds, aromatic hydrocarbons, and cyanides.

3.4 The material shall also conform to the requirements given in Table 1.

3.5 Description of Alumina

Alumina used in colour (dried as aluminium hydroxide) is a white, odourless, tasteless, amorphous powder consisting essentially of aluminium hydroxide ($Al_2O_3 . H_2O$).

3.5.1 *Acidity or Alkalinity*

Agitate 1 g of alumina with 25 ml of water and filter, the filtrate shall be neutral to litmus paper.

3.5.2 Alumina used in colour shall also conform to the requirements given in Table 2.

Table 1 Requirements for Aluminum Lake of Sunset Yellow, FCF
(*Clause* 3.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Sunset yellow dye used in preparation of lake	Shall conform to specification laid down in IS 1695	—	IS 1695
ii)	Pure dye content of aluminium lake, percent by mass, *Min*	17	A	—
iii)	Chlorides and Sulphates expressed as sodium salt, percent by mass, *Max*	2	B and C	—
iv)	Inorganic matter insoluble in HCl, percent by mass, *Max*	0.5	D	—
v)	Aluminium content in the lake, percent by mass, *Max*	44	E	—
vi)	Lead, mg/kg, *Max*	10	—	**15** of IS 1699
vii)	Arsenic, mg/kg, *Max*	3	—	**15** of IS 1699

Table 2 Requirements Alumina Used in Colour

(*Clause* 3.5.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of this Standard	Cl of IS 1699
i)	Lead, mg/kg, *Max*	10	—	**15**
ii)	Arsenic, mg/kg, *Max*	1	—	**15**
iii)	Mercury (as Hg), ppm, *Max*	1	—	**15**
iv)	Aluminium hydroxide (Al_2O_3), percent by mass, *Min*	50	F	—

3.6 The material shall be processed, packed, stored and distributed under hygienic conditions in licensed premises (*see* IS 2491).

4 PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in metal containers, polyethylene containers, or cardboard containers suitably lined with polyethylene. Any other suitable containers may also be used, subject to agreement between the purchaser and the vendor.

4.2 Marking

4.2.1 Each container shall be legibly and indelibly marked to give the following information:

a) The words 'Food Colour';
b) Common name of the colour;
c) Chemical name of the colour;
d) Colour index number;
e) Source of manufacture;
f) Date of manufacture;
g) Net quantity, in g or kg;
h) Batch or Code No.; and
j) Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules*, 2011 and *Food Safety and Standards (Packaging and Labelling) Regulation,* 2011 and the Rules framed thereunder.

4.2.2 *BIS Certification Marking*

The product may also be marked with the Standard Mark.

4.2.2.1 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 Representative samples of the material for tests shall be drawn and criteria for ascertaining conformity to the requirements of this standard shall be determined according to the method prescribed in **4** of IS 1699.

5.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the experimental results.

ANNEX A

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF PURE DYE CONTENT IN ALUMINIUM LAKE OF SUNSET YELLOW

A-1 APPARATUS

A-1.1 U.V. Visible Spectrophotometer Lambda 20.

A-1.2 Matched pair of 10 mm path length optical cells.

A-1. 3 All volumetric glassware shall be of A grade quality.

A-2 PROCEDURE

Weigh accurately 250 mg of the Lake sample into a tarred butter paper and transfer into a 250 ml volumetric flask. Add 50 ml distilled water and 5 ml hydrochloric acid (HCl). Heat the flask on hot plate until the sample appears to be dissolved. Remove the flask from hot plate and allow it to cool to room temperature. Dilute to volume with distilled water and shake to mix. If the solution is cloudy from an insoluble lake substratum, filter approximate 50 ml through a Buckner funnel discarding the first 10 ml of filtrate. Pipette, 1 ml of this solution and dilute to 100 ml with distilled water, to make a final concentration of 1 mg/100 ml. Determine the maximum absorbance (ABS) of this solution at the respective wavelength against distilled water as blank, in a glass cell with 10 mm light path.

NOTE — Apply normal precautions while handling general laboratory reagents. Wear goggles for eye protections.

A-3 CALCULATION

$$\text{Pure dye content in the sample, percent by mass} = \frac{ABS \times 100}{M \times 543}$$

where

ABS = maximum absorbance found;

M = mass of the sample, in g in 100 ml solution; and

543 = $E^{1\%}$ cm 482 nm, for sunset yellow FCF.

ANNEX B

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF CHLORIDE IN ALUMINIUM LAKE OF SUNSET YELLOW

B-1 APPARATUS

Potentiometer titration apparatus with silver indicator electrode and glass reference electrode.

B-2 REAGENTS

B-2.1 Silver Nitrate Solution — 0.1N. (Dissolve 1.700 g of silver nitrite in 25 ml distilled and dilute to 100 ml with distilled water.)

B-2.2 Nitric Acid Solution — 1.5 N. [Dilute 9.7 ml of conc. HNO_3 (69 percent) to 100.0 ml with distilled water].

B-3 PROCEDURE

B-3.1 Weigh accurately 10 g of the sample. Add 250 ml distilled water. Stir to wet out the sample and then stir occasionally during 30 min. Filter through Whatman filter paper. Measure 50 ml of the filtrate, dilute to 300 ml with water and acidify with 5 ml of 1.5 N Nitric acid solution. Place the silver electrode and glass electrode in the dye solution and connect to the potentiometer titration. Determine the chloride content of the solution by titration against the 0.1N silver nitrate solution and calculate the result as sodium chloride. One ml of 0.1 N silver nitrate ($AgNO_3$) solution = 0.005 85 g of sodium chloride (NaCl). Express the result as a percentage of the mass of sample taken.

B-4 CALCULATION

$$\text{Chloride content, percent} = \frac{\text{Volume of 0.1N } AgNO_3 \times 0.005\,85 \times 100}{\text{Weight of sample}}$$

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF SULPHATE IN ALUMINIUM LAKE OF SUNSET YELLOW

C-1 REAGENTS

C-1.1 Hydrochloric Acid — 1.16 to 1.18.

C-1.2 Barium Chloride Solution — 0.25 N.

C-2 PROCEDURE

C-2.1 Weigh accurately 10 g of the sample. Add 250 ml distilled water. Stir to wet out the sample and then stir occasionally during 30 min. Filter through Whatman filter paper. Measure 50 ml of the filtrate, dilute to 300 ml with water and acidify with hydrochloric acid, adding 1 ml in excess. Heat the solution on a water bath and add an excess of 0.25 N barium chloride solution drop by drop, with stirring. Heat the solution on water bath for 4 h and allow the precipitate to settle or leave it overnight at room temperature. Filter the solution through previously weighed sintered glass crucible and wash with hot distilled water. Dry the crucible in the oven for 1 h at 135°C. Allow to cool in a desiccator for 30 min and reweigh.

C-3 CALCULATION

$$\text{Sodium sulphates, percent} = \frac{W_1 \times 0.608\,6 \times 2.5 \times 100}{W_2}$$

where

W_1 = weight of $BaSO_4$ ppt, in g; and
W_2 = weight of sample, in g.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF INORGANIC MATTER INSOLUBLE IN HCl IN ALUMINIUM LAKE OF SUNSET YELLOW

D-1 APPARATUS

Sintered glass filters.

D-2 REAGENTS

D-2.1 Concentrated Hydrochloric Acid [HCL]

D-2.2 Hydrochloric Acid, 1 N.

D-3 PROCEDURE

D-3.1 Weigh accurately about 1.0 g of sample in a 500 ml beaker, add 300 ml of distilled water and conc. HCl (20 ml) to dissolve the sample. Heat the solution in a fume hood at 80-90°C and filter this solution in hot condition through previously weighed crucible.

Wash the crucible with 1N HCl until the washing is colourless. Dry the crucible at 135°C for 3 h. Cool in a desiccator and weigh.

NOTE — Safety gloves and glasses must be worn when handling concentrated acids.

D-4 CALCULATIONS

$$\text{HCl insoluble matter} = \frac{(M_2 - M_1) \times 100}{M}$$

where

M_2 = mass of the sintered glass crucible with residue, in g;
M_1 = mass of the crucible, in g; and
M = mass of the sample taken, in g.

ANNEX E

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF ALUMINIUM CONTENT IN THE ALUMINIUM LAKE OF SUNSET YELLOW

E-0 GENERAL

Titanous chloride (titanium trichloride, $TiCl_3$) reduces the colouring matter to yield titratable reduction products. The method assumes that isomers and subsidiary colouring matters have the same titanium trichloride equivalent as the main colouring matter.

E-1 APPARATUS

E-1.1 Titration Apparatus

E-1.1.1 *Bottle* (borosilicate glass) for titrant (may be up to 5 litre volume, as needed), with 29/42 ground glass center neck (for burette), side arm for inlet gas sparge, side arm stopcock (for gas outlet), and side arm with glass stopper for refilling bottle

NOTE — Bottle may need to be custom-made.

E-1.1.2 *Digital Burette*, 25 ml — Digital burette or equivalent to 500 ml conical flasks, sealable with No.10 rubber stoppers.

E-1.1.3 *Overhead Stirrer*

E-1.1.4 *Stopper Assembly* — No. 10 rubber stopper with five holes for accommodating the stirrer rod, burette delivery tip, argon source, gas outlet tubing, and 10 ml pipette.

E-1.1.5 *Glass rod stopper* for the pipette inlet in the stopper assembly.

E-1.1.6 *Tubing,* glass and flexible plastic, for connections.

E-1.1.7 *Hot Plate*

E-2 REAGENTS

E-2.1 Titanium Trichloride (20 percent in HCl), reagent grade.

E-2.2 Hydrochloric Acid, reagent grade.

E-2.3 Ferrous Ammonium Sulphate [Fe $(NH_4)2(SO_4)_2.6H_2O$], reagent grade.

E-2.4 Sulphuric Acid, reagent grade.

E-2.5 Potassium Dichromate, 0.100 N standard solution.

E-2.6 Ammonium Thiocyanate, reagent grade.

E-2.7 Sodium Citrate, reagent grade.

E-2.8 Sodium Hydrogen Tartrate, reagent grade.

E-2.9 Boiling Chips

E-2.10 Argon, UHP Compressed Gas (Carbon dioxide from a Kipp apparatus may be also be used, but is much less convenient; compressed nitrogen gas may be used provided residual oxygen is removed.)

NOTE — A water bubbler should be placed in line between the argon source and the titration apparatus.

E-2.11 Preparation of 0.1 N Titanium Trichloride

Measure 800 ml of water for each litre of solution required into a beaker of appropriate size. On a hot plate, boil the water vigorously for 1 min, cover with a watch glass, and allow to cool to room temperature. In a fume cupboard, using graduated cylinders, add 90 ml of hydrochloric acid, stir, and add 100 ml of 20 percent titanium trichloride solution, for each litre of solution required. (Avoid transferring any white precipitate from the titanium trichloride reagent bottle). Mix the solution and transfer to the titrant bottle. Attach the burette and connect the argon. Pass argon through the solution for 1-2 h with the sidearm stopcock on the bottle open to maintain ambient pressure. While maintaining a slow flow of gas, draw up titrant into the burette. Drain the burette, discard the titrant, and refill. Drain and refill the burette two more times. Stop the gas flow, close the sidearm stopcock, and store the solution for at least 72 h before use.

E-2.12 Standardization of 0.1 N Titanium Trichloride

Drain and refill the burette with 0.1 N titanium trichloride. Use within 1 h. Weigh 3.0 g (0.2 g) of ferrous ammonium sulphate into a 500 ml conical flask. Add 200 ml of water. Using a graduated cylinder, add 25 ml of 10 N sulphuric acid. Pipette 20 ml of standard 0.100 N potassium dichromate into the flask. Swirl to mix. Connect the flask securely to the stopper assembly (rubber stopper fitted with stirrer, gas inlet and outlet tubes, burette tip, and glass rod stopper). Gently bubble argon into the flask. Turn on the stirrer and slowly increase the speed until the solution is stirring vigorously without splashing. Wait 1 min before beginning the titration, and continue stirring throughout the procedure.

After adding 15 -17 ml of 0.1 N titanium trichloride drop-wise within about 2 min, stop the flow of titrant and reduce the argon flow. Remove the solid glass rod from the stopper assembly, and pipet 10 ml of 50 percent ammonium thiocyanate (indicator solution) into the flask. The colour of the solution will become brownish-red. Remove the pipette, re-insert the glass rod, and restore the argon flow. Add 0.1 N titanium trichloride drop wise, with 2-3 s pauses between drops, until a sharp colour change from brownish-red to light green is observed. The endpoint (20-21 ml) is reached when the solution returns to the original light green colour and remains that colour for 20 s. Stop the argon flow and gradually turn off the stirrer. Record the volume (*V*) of 0.1 N titanium trichloride used to the nearest 0.05 ml. Perform the titration procedure in triplicate.

Determine the indicator blank by repeating the above procedure without the 0.100 N potassium dichromate. The blank determination should require less than 0.5 ml of 0.1 N titanium trichloride. Record the volume used to the nearest 0.05 ml.

Calculate the concentration of the standard titanium trichloride solution by averaging the three titration results. (Restandardize the solution weekly by performing one titration of 0.100 N potassium dichromate and determining one indicator blank.)

$$\text{For each titration, the concentration of the titanium trichloride solution} = \frac{(N \times 20)}{(V - VB)}$$

where

N = concentration of the standard potassium dichromate solution;

20 = aliquot (ml) of the potassium dichromate solution;

V = volume (ml) of titanium trichloride solution required to titrate the aliquot of standard potassium dichromate solution; and

VB = volume (ml) of titanium trichloride solution used to determine the blank.

E-3 PROCEDURE

Add 150 ml of water to a 500 ml conical flask and dissolve in it 10 g of sodium citrate. Accurately weigh a quantity of lake equivalent to 35-40 ml of 0.1 N titanium trichloride and transfer it to the flask. Add a few boiling chips, wash down the walls of the flask with water, and cover with a watch glass. In a fume hood, heat the mixture to boiling or until the lake has completely dissolved. Using gloves, remove the flask from the hot plate. Within 2-4 min of removing the flask from the hot plate, remove the watch glass, and connect the flask securely to the stopper assembly (flask might still be hot). Gently bubble argon into the flask. Turn on the stirrer and slowly increase the speed until the solution is stirring vigorously without splashing. Wait 1 min before beginning the titration, and continue stirring throughout the procedure. The colour will act as its own indicator unless otherwise stated in the appropriate monograph. Rapidly add standardized 0.1 N titanium trichloride drop wise until the colour of the solution begins to change, then stop for 15-20 s. Continue adding the titrant dropwise, with 1-2 s pauses between drops. When the solution is close to the final colour, stop again for 20 s. Continue adding 0.1 N titrant drop wise, with 5-10 s pauses between drops, until the final colour is observed. The endpoint is reached when the final colour is stable for 20 s. Stop the argon flow and gradually turn off the stirrer. Record the volume of titrant used to the nearest 0.05 ml.

E-4 CALCULATION

$$\text{The percent total colouring matters content of the sample} = \frac{100 \times (V \times F \times N)}{W}$$

where

V = standardized titanium trichloride solution required, in ml;

F = D / (1.00 ml × 0.1 meq/ml), where D is the weight (mg) of colouring matters equivalent to 1.00 ml of 0.1 N titanium trichloride, that is 11.31 mg);

N = concentration of standardized titanium trichloride solution (in meq/ml); and

W = weight of the sample taken, in mg.

ANNEX F

[*Table* 2, *Sl No.* (iv)]

DETERMINATION OF ALUMINUM HYDROXIDE IN ALUMINA

F-0 PRINCIPLE

Water-soluble aluminates are determined by precipitating the material with ammonium hydroxide twice, igniting and weighing as R_2O_3 The mass of oxides due to iron and titanium is deducted from it to calculate the mass of Al_2O_3.

F-1 REAGENTS

F-1.1 Ammonium Chloride — Solids.

F-1.2 Concentrated Nitric Acid — As per IS 264.

F-1.3 Dilute Ammonium Hydroxide — Approximately 9 percent.

F-1.4 Dilute Hydrochloric Acid — 1: 4 *(v/v)*.

F-1.5 Ammonium Nitrate Solution — Approximately 2 percent (made neutral with ammonium hydroxide to methyl red or phenol red).

F-1.6 Methyl Red Indicator Solution — 0.15 g in 500 ml of water.

F-2 PROCEDURE

F-2.1 Take 25 ml of the prepared solution and dilute to 100 ml. Add 5 g of ammonium chloride and 3 to 4 ml of nitric acid. Boil the solution for a few min. Add 2 to 3 drops of methyl red solution. Add ammonium hydroxide drop wise, with stirring, until the colour of the solution changes to distinct yellow. Boil for 1 min, filter through a filter paper (Whatman No. 41 or its equivalent) and wash the precipitate three times with hot ammonium nitrate solution. Transfer the precipitate to the original beaker washing the filter paper with the dilute hydrochloric acid.

F-2.2 Add hot dilute hydrochloric acid sufficient to dissolve the precipitate and boil. Add methyl red indicator and make it faintly ammoniacal as before. Boil again for 1 min and filter the precipitate as before. Wash the precipitate with hot ammonium nitrate solution until free from chlorides and transfer it with the filter paper to a crucible, previously ignited to constant mass. Dry and ignite at a low temperature until all carbon is burnt off; then complete the ignition at 1 100°C for 1 h. Cool the residue in a desiccator over a good desiccant and weigh. Re-ignite it until constant mass is attained.

F-2.3 Determine the percentage of aluminium oxide.

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Amendments Issued Since Publication

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.org.in

Regional Offices:		*Telephones*
Central	Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002	2323 7617 2323 3841
Eastern	1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi KOLKATA 700054	2337 8499, 2337 8561 2337 8626, 2337 9120
Northern	SCO 335-336, Sector 34-A, CHANDIGARH 160022	260 3843 260 9285
Southern	C.I.T. Campus, IV Cross Road, CHENNAI 600113	2254 1216, 2254 1442 2254 2519, 2254 2315
Western	Manakalaya, E9 MIDC, Marol, Andheri (East) MUMBAI 400093	2832 9295, 2832 7858 2832 7891, 2832 7892

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